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Studying the Effect of Phosphogypsum Addition on Ceramic Membrane Properties

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HIGHLIGHTS

- Possibility of use of phosphogypsum as additive in ceramic membrane
- Effects of phosphogypsum on ceramic membrane properties.
- Adding of phosphogypsum leads to increased hydrophilicity, decreased mechanical strength, and increased porosity of ceramic membranes.
- phosphogypsum has the potential to significantly affect ceramic membrane properties and its use in various membrane applications.

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ABSTRACT

This study aimed to investigate the effects of phosphogypsum addition on ceramic membrane properties. In this regard, clay and phosphogypsum were characterized using FTIR and laser particle sizer. The ceramic paste was prepared by incorporating varying amounts of phosphogypsum (10 to 50%), followed by molding using a semidry-pressing process at 10 bars pressure and sintering temperature at 900°C. The raw materials and prepared membranes were analyzed using FTIR, laser particle size, contact angle, porosity, and mechanical strength to evaluate the properties of the resulting ceramic membranes. The study showed that as the amount of phosphogypsum increased from 10% to 50%, the membrane's hydrophilicity significantly increased, while its mechanical strength decreased by 35% and porosity increased by 26%. Moreover, the permeability of distilled water also showed a significant increase of 67% when the amount of phosphogypsum was increased from 10% to 50%.These observations suggest that phosphogypsum can significantly influence ceramic membrane properties, which may have implications for its use in various membrane applications. The findings of this research contribute to our understanding of the potential use of phosphogypsum as a valuable material for ceramic membrane production, with important implications for sustainable waste management practices. Future studies can focus on exploring the suitability of these ceramics for various applications and their environmental impact.

1. Introduction

Membrane technology is a rapidly evolving field that has revolutionized the separation and purification processes in various industries [1], including principally wastewater treatment and water distillation [2,3], pharmaceuticals [4,5], food and beverage [6–8], and biotechnology [9,10]. Membranes can be produced from polymeric or ceramic materials. Polymeric membranes are widely used in various applications due to their ease of production, high permeability, and low cost [11]. On the other hand, ceramic membranes are known for their superior mechanical and chemical stability, high selectivity, and excellent performance under harsh conditions [12,13]. Many configurations can be remarked for membrane modules such as flat, hollow fiber, helical, and spiral [14,15].

To enhance the performance of ceramic membranes, a variety of additives can be utilized during their preparation in conjunction with clay. These additives can have a significant impact on crucial properties, including porosity, mechanical strength, stability, permeability, and selectivity. Of these properties, porosity has been a primary focus of additive research, with the use of waste materials as an effective strategy for enhancing membrane porosity. For example, the incorporation of oasis waste at concentrations of 8 to 15% has been shown to increase porosity by up to 54% [16]. Similarly, other studies have

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successfully employed corn cob ash and starch as additives to enhance membrane porosity [17,18]. Achiou et al. [19] reported that the addition of starch in concentrations ranging from 0 to 20 wt% resulted in a remarkable 68% increase in porosity, reaching a value of 49.7%. These findings highlight the potential of waste materials and natural additives to enhance porosity and other properties of ceramic membranes, with important implications for their use in various industrial applications.

The brittleness of ceramic membranes remains a major challenge in their practical applications. To address this issue, various modifications have been investigated, and among them, the addition of ferric oxide (Fe2O3) has shown promising results in enhancing mechanical strength, membrane formation, and surface morphology. Previous studies have reported that incorporating Fe_2O_3 from 0 to 10% leads to a 14% increase in mechanical strength [20]. Similarly, Li et al. [21] have demonstrated that increasing Fe_2O_3 content from 8 to 12% resulted in a remarkable 27% improvement in mechanical strength, reaching a value of 1.92 MPa. However, a further increase in Fe_2O_3 content from 12 to 25% led to a decrease in mechanical strength, which dropped to 1.36 MPa. These findings highlight the potential of Fe_2O_3 as an effective strategy to enhance the mechanical properties of ceramic membranes while also pointing out the importance of optimizing the Fe_2O_3 concentration for achieving optimal performance.

To enhance the permeability of ceramic membranes, researchers have explored the use of waste materials. One promising approach has been to incorporate Coal Fly Ash into the production process. This technique has yielded membranes with both low cost and high permeability, as well as effective filtration capabilities for wastewater [22]. A study by Al-Shaeli et al. [23], further supports the use of waste additives for ceramic membrane production, as it has been shown to improve mechanical strength, chemical stability, selectivity, and overall permeability.

Numerous additives have been proposed; among them, we find phosphogypsum, an industrial waste product that results from the wet process used in phosphorus fertilizer production. According to Bounaga et al. [24], five tons of phosphogypsum are generated for every ton of P_2O_5 produced, resulting in over 300 million tons of annual global phosphogypsum production [25] of which 5000 tons were generated in Gabes-Tunisia phosphate fertilizer plant [26]. To address these concerns, researchers have explored various valorization approaches for phosphogypsum, such as incorporating it as a reinforcing agent in geopolymer cement to improve their mechanical and microstructural properties [27], using it as a road material [28,29], as a thermal storage material [30].

In this study, we investigated the effects of adding phosphogypsum to clay for membrane preparation. To the best of our knowledge, no prior research has examined this topic. We carefully analyzed the properties of the phosphogypsum-clay membranes, including their contact angle, porosity, mechanical strength, and permeability.

2. Material and Methods

2.1 Clay-Phosphogypsum Membrane Preparation

The objective of this research was to fabricate ceramic membranes through the utilization of a mixture of clay and phosphogypsum. The clay sample was collected from the Matmata region in Gabes-Tunisia. The raw materials were ground to uniform particle sizes ranging from 80 to 160 µm using a Matest A092 brand grinder, and their particle size distribution was determined. Subsequently, the clay and phosphogypsum were combined in predetermined ratios until achieving homogeneity. Distilled water (22%) was added to the dry mixture to produce a moldable paste. To prevent the formation of air bubbles and membrane cracks during the sintering process, the paste was treated with ultrasonic waves for 30 minutes. After resting at room temperature for 24 hours, the paste was pressed into cylindrical samples of 3 mm thickness and 55 mm diameter using an Edibon EEU/20KN press under 10 bars. Following pressing, the samples were sintered at 900°C in a Nabertherm laboratory furnace.

2.2 Particle Size Distribution and Chemical Characterization

To ensure the desired morphological characteristics of the ceramic membrane, it is crucial to determine the appropriate granulometry of the mineral powder. The particle size distribution of both clay and phosphogypsum was analyzed using a laser diffraction particle size analyzer (Malvern Mastersizer 3000E). Fourier transforms infrared (FTIR) analysis was conducted to chemically characterize the samples, using a Spectrum Two FTIR spectrometer with a diamond attenuated total reflectance (ATR) accessory. Spectra were recorded with a spectral resolution of 2 cm^{-1}, averaging four scans in the wavenumber range of 450-4000 cm^{-1}.

2.3 Membrane Contact Angle

We employed the KRÜSS Drop Shape Analyzer - DSA25 to assess the hydrophobic or hydrophilic nature of the membrane using contact angle measurements. The contact angle was determined with the assistance of the Advance software. This allowed us to obtain precise and accurate measurements of the membrane's surface characteristics, which are crucial for understanding its performance in various applications.

2.4 Membrane Porosity

To investigate the impact of phosphogypsum addition on the porosity of a ceramic membrane, the Archimedes method was employed to determine the porosity of the membrane [31,32]. To conduct this analysis, the ceramic membrane was first dried in a Memmert oven UN 30 at 105°C until a constant weight was achieved (m_1, kg). Next, the membrane was placed in a container, and we used a vacuum pump to reduce the pressure inside the container for 30 minutes. The membrane was then

immersed in water for 15 minutes before being removed, gently dried, and weighed (m_2, kg). The open porosity (OP) of the membrane was then calculated using the equation below:

$$OP = \frac{m_2 - m_1}{\rho_{water} V_m} \quad (1)$$

With ρ_{water} is the water density (kg m^{-3}), and V_m is the membrane volume (m^3).

2.5 Mechanical Strength

We performed mechanical strength tests using the Edibon EEU/20KN Universal Material Testing Unit. This equipment allowed us to evaluate the mechanical properties of the ceramic membrane and to see the effect of the addition of phosphogypsum on the mechanical resistance of the ceramic membranes.

2.6 Membrane Permeability

To evaluate the permeability of the clay-phosphogypsum membrane, we employed a laboratory apparatus, as illustrated in Figure 1. The membrane was affixed at the unit's base and subjected to a flow of distilled water under nitrogen gas pressure. By monitoring the mass of the permeate as a function of time, we were able to establish the membrane's permeability.

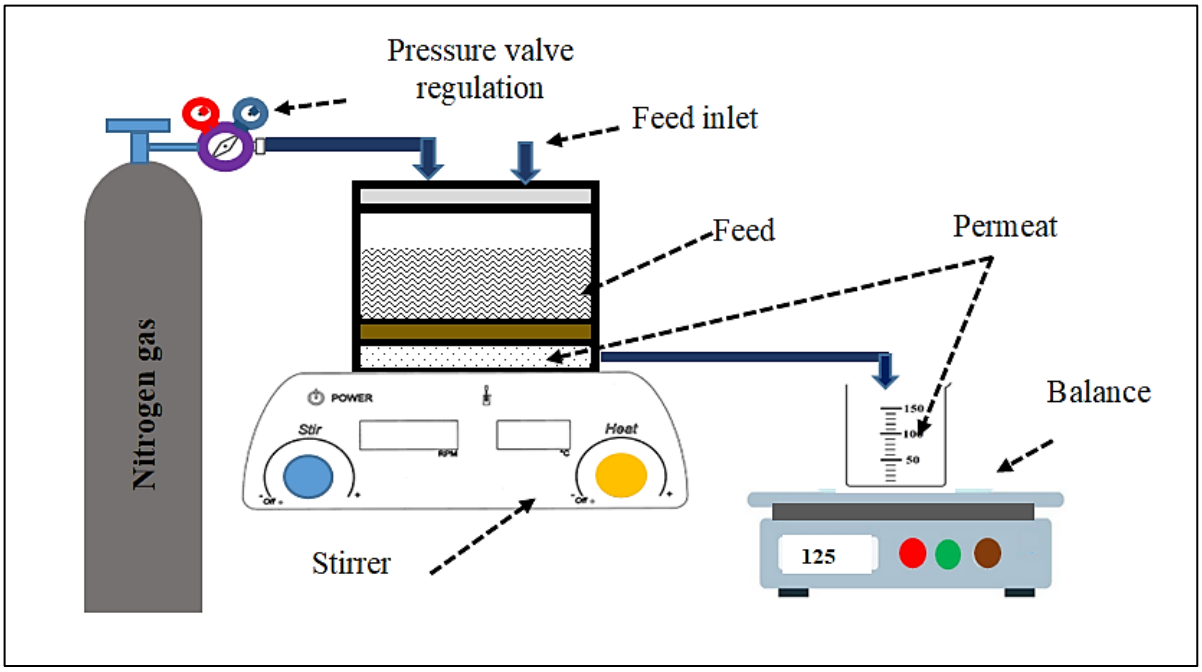


Figure 1: Set-up of the dead-end installation

3. Results and Discussion

3.1 FTIR of Clay

The peaks at 3694 and 3620 cm^{-1} Figure 2 were assigned to the stretching vibration of the hydroxyl groups (OH) of illite and kaolinite [16,33]. The peak assigned to H–O–H and OH bending of water can be shown at around 1635 cm^{-1}. The absorption band at 1449 cm^{-1} is related to calcite vibration [34]. The peak attributed to symmetrical and asymmetrical elongation vibrations of the Si-O-Si occurs at 1026 cm^{-1} [35]. In addition, the peak obtained at around 919 cm−1 suggests Al-OH deformation vibration [36]. Also, the peaks at around 875, 774, 690, 517, and 466 cm^{-1} are attributed to the deformation of OH linked to Al^{3+} and Fe^{3+}, the vibration of groups MgO and Si–O–Al, Si–O–Si bending vibrations, stretching vibrations and elongation of Si-O-Al, [37–39].

3.2 FTIR of Phosphogypsum

Figure 3 displays the FTIR spectrum of phosphogypsum. We consulted the works of Agayr et al. [40], Zemni et al. [41], Bouargane et al. [42], Mechi et al. [43], and Hammas-Nasri et al. [44] to assign the FT-IR peaks. The observed bands at 3504–3393 cm−1 and 1682–1618 cm−1 were attributed to the stretching and deformation vibrations of OH^- and H-O-H groups from water molecules, respectively, as depicted in the figure. The large band represented the stretching vibration of SO_4^{2-} at 1096 cm^{-1}. The deformation vibration of SO_4^{2-} observed at 666-597 cm^{-1}.

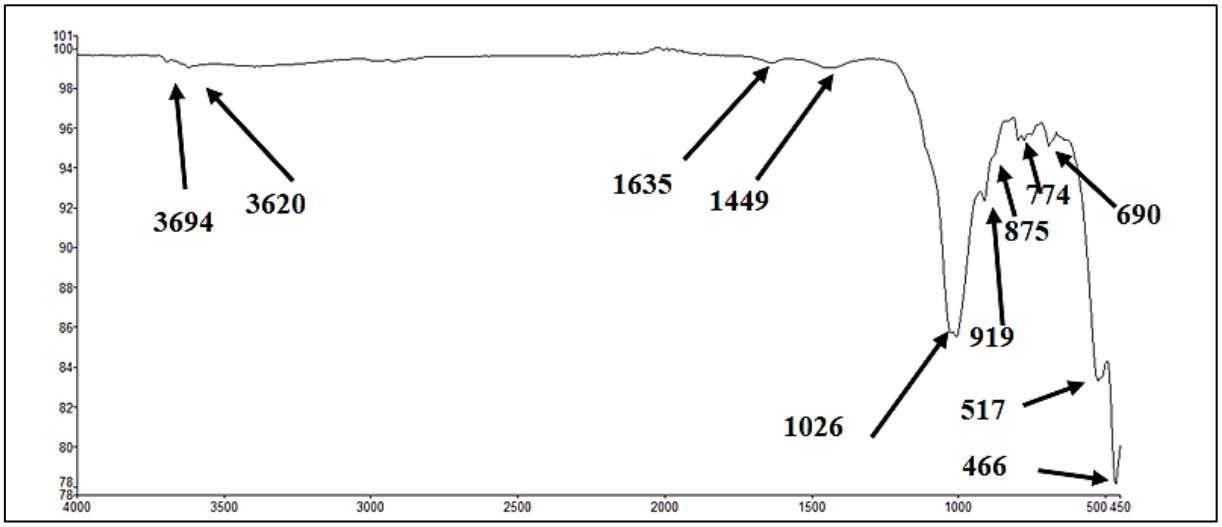


Figure 2: FTIR spectrum of the clay powder

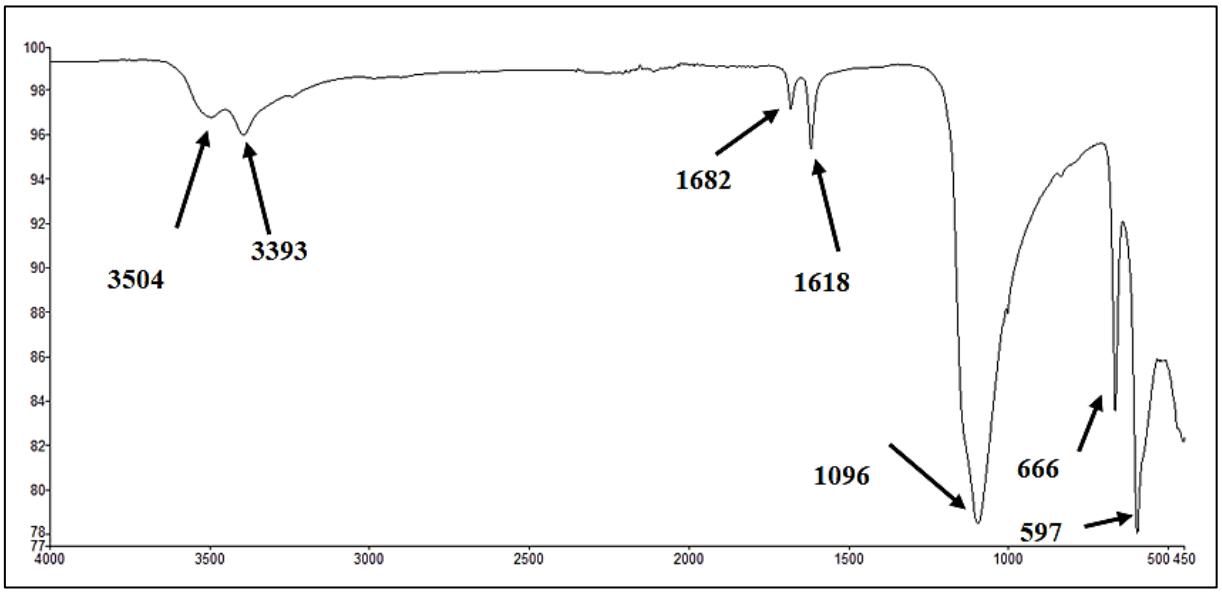
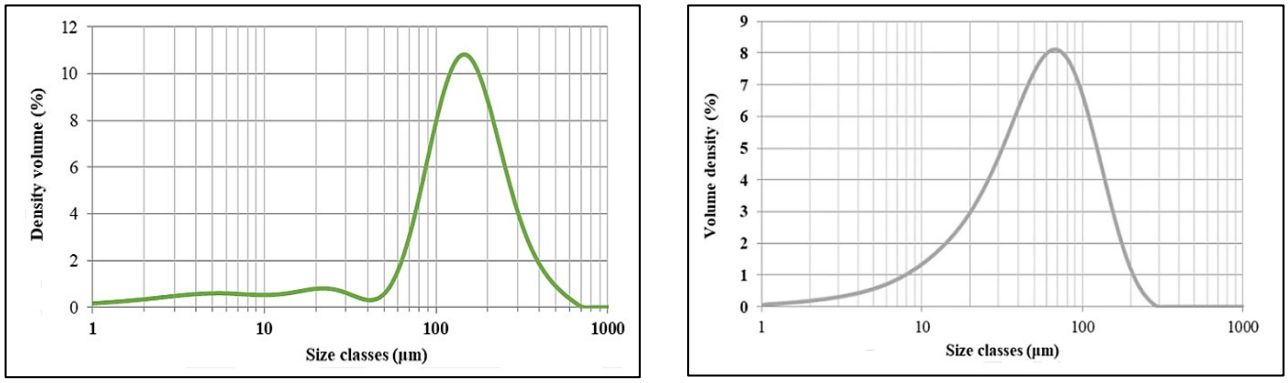


Figure 3: Phosphogypsum FTIR spectrum

3.3 Particle Size Distributions of Clay and Phosphogypsum

Yanu et al. [45], Khemakhem et al. [46], and Aloulou et al. [47] established that the optimal particle size for the development of a microfiltration ceramic membrane falls within the 100 µm range. In our study, the particle size distribution of both clay and phosphogypsum are depicted in Figure 4, and we observe that our particle sizes fall within the aforementioned optimal range.



(a) (b)

Figure 4: Granulometric Distribution Of (a) clay particles and (b) phosphogypsum particles

3.4 Effect of Phosphogypsum on Membrane Contact Angle

The contact angle measures the degree of wetting of a surface, and changes in contact angle can indicate changes in surface hydrophobicity or hydrophilicity. Therefore, investigating the effect of phosphogypsum on the ceramic membrane contact angle can provide valuable information on its impact on ceramic membrane properties and its potential application in various fields. For this case, we have varied the amount of phosphogypsum in the ceramic membrane from 10 to 50%. The obtained results are plotted in Figure 5. According to this figure, the addition of phosphogypsum to the ceramic membrane potentially alters its surface properties, leading to changes in its hydrophobicity or hydrophilicity, which can be reflected in the contact angle of the ceramic membrane. The values of the contact angle of the prepared membranes are below 90°, and these membranes present a hydrophilic comportment. With the increase of the phosphogypsum amount in the ceramic membrane, a decrease of about 66% of the contact angle can be remarked. This decrease can be explained by the fact that the contact angle is influenced by the chemical composition and physical properties of the surface. Incorporating phosphogypsum, a mineral compound, to the membrane surface may introduce new functional groups, potentially resulting in a reduction of the contact angle and an increase in the surface energy of the membrane.

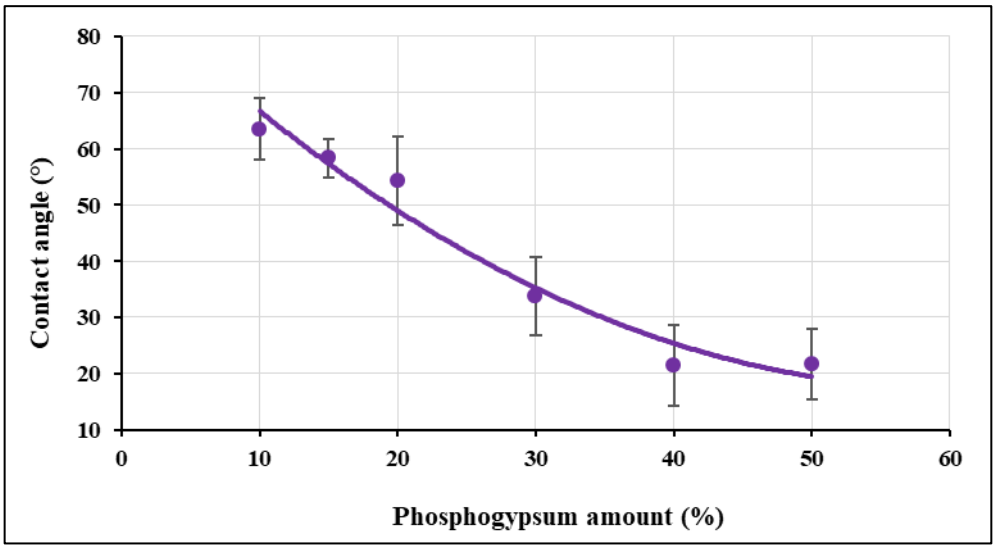


Figure 5: Contact angle VS phosphogypsum amount

3.5 Effect of Phosphogypsum on Membrane Porosity

Porosity is a critical parameter that directly influences a membrane's filtration performance, affecting both its flow rate and selectivity. The effects of adding phosphogypsum on membrane porosity are presented in Figure 6. According to this figure, porosity increases with the increase in the amount of phosphogypsum. This evolution can be explained by the fact that phosphogypsum, which contains high levels of calcium sulfate, can serve as a pore-forming agent [48–50] during membrane fabrication. When uniformly dispersed throughout the ceramic membrane, calcium sulfate particles can decompose upon heating, leaving voids within the membrane structure. These voids can enhance the overall porosity of the membrane and create a more interconnected pore structure. Furthermore, phosphogypsum can lower the sintering temperature of the ceramic particles in the membrane, leading to an increase in porosity [51]. Sintering involves heating and fusing ceramic particles to form a solid membrane structure. The inclusion of phosphogypsum can reduce the sintering temperature required for the particles to fuse, resulting in a more porous and open structure for the membrane.

3.6 Effect of Phosphogypsum on Mechanical Strength

Figure 7 shows the evolution of membrane mechanical strength as the composition of phosphogypsum changes. The data suggests that there is a negative correlation between the amount of phosphogypsum in the membrane and its mechanical strength. Specifically, as the composition of phosphogypsum increases from 10 to 50%, the mechanical strength of the membrane decreases from 2.8 to 1.8 MPa. The highest mechanical strength of 2 is observed at the lowest phosphogypsum composition of 10%, while the lowest mechanical strength of 1.8 MPa is observed at the highest phosphogypsum composition of 50%. As previously discussed, an increase in porosity was obtained for the higher amount of phosphogypsum added to the ceramic membrane. The porosity in the ceramic membranes contributes to an increase in the stresses during the compression tests, which will reduce the resistance of the ceramic products. The same finding has been reported by Rahim et al. [52]. This information can be valuable for designing and optimizing the composition of membranes, particularly in applications where mechanical strength is an important consideration, such as in water treatment or gas separation processes. The results suggest that reducing the amount of phosphogypsum in the membrane can lead to an increase in its mechanical strength.

Phosphogypsum can introduce voids or defects into the membrane structure, which can increase its porosity. The formation of these voids depends on factors such as the amount of phosphogypsum added, the sintering temperature, and the composition of the ceramic particles.

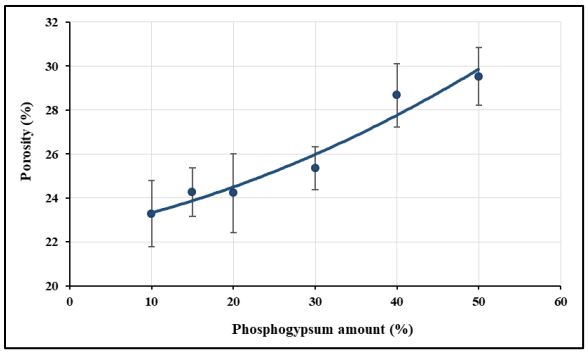


Figure 6: Evolution of membrane porosity with phosphogypsum

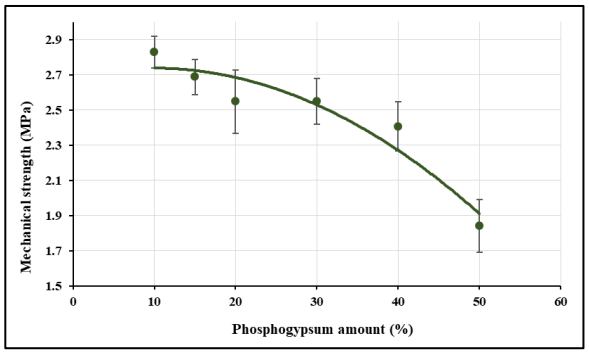


Figure 7: Effect of phosphogypsum on mechanical strength

3.7 Effect of Phosphogypsum on Membrane Permeability

Figure 8 depicts the correlation between the water permeability of clay-phosphogypsum and the quantity of phosphogypsum employed. Our findings demonstrated a noteworthy 67% enhancement in permeability when the phosphogypsum proportion varied from 10% to 50%. The rise in permeability is primarily due to the observed amplification in porosity and hydrophilicity, as illustrated in Figure 5 and Figure 6, respectively. The escalation in porosity and hydrophilicity generates a greater permeate flux, ultimately leading to an increase in permeability.

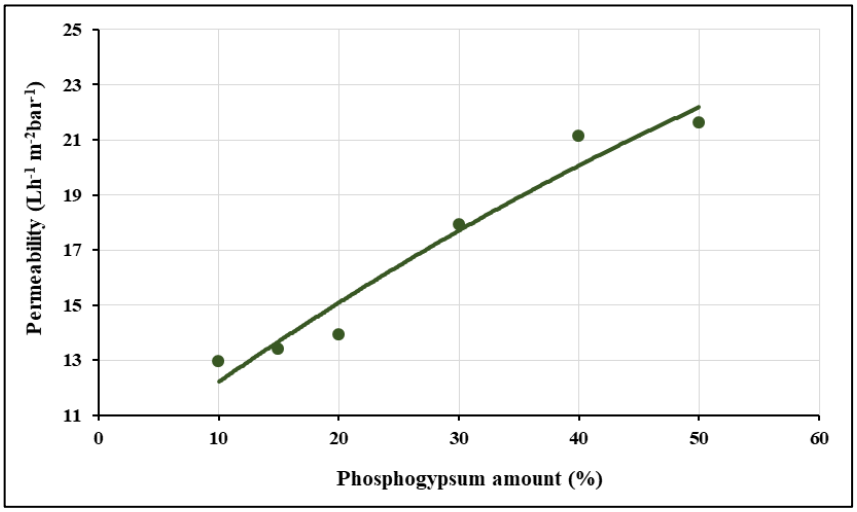


Figure 8: Variation of membrane permeability with phosphogypsum amount

4. Conclusion

This study used the semidry-pressing process to prepare and characterize ceramic membranes with varying amounts of phosphogypsum (10-50%). The results revealed that phosphogypsum had a significant impact on the properties of ceramic membranes. Increasing the amount of phosphogypsum led to a 66% increase in membrane hydrophilicity, indicating its potential use in the microfiltration of wastewater. Furthermore, the addition of phosphogypsum resulted in a 67% increase in permeability and a 26% increase in porosity, which led to a 35% decrease in mechanical strength.

Future studies can focus on exploring the suitability of these clay-phosphogypsum membranes for various applications and their environmental impact. Overall, this study provides valuable insights into the potential use of phosphogypsum in developing ceramic membranes with improved properties, which could have significant implications for developing sustainable technologies for water treatment and environmental remediation.

Author contribution

All authors contributed equally to this work.

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Data availability statement

The data that support the findings of this study are available on request from the corresponding author.

Conflicts of interest

The authors declare that there is no conflict of interest.

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